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AEROJET-GENERAL CORPORATION SPACE PHYSICS DEPARTMENT

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CRYOGENIC-SOLID COOLING TECHNIQUES

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ABSTRACT

Evaluation of the experimental model of the cryogenic-solid cooling system, with subsequent modification and testing, is reported herein. Advancements in the investigation of cryogenic-coolant solidification techniques and continued efforts in the development of temperature and pressure control methods are also described. Conclusions and recommendations are made, based upon the progress of work performed to date.

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I. INTRODUCTION

The object of this program is to evolve the specific techniques for making a laboratory model, cryogenic-solid cooling system, and for measuring the physical and thermodynamic parameters of the operating device. The results of the second three months of investigation are summarized in this report.

This period was spent primarily in evaluating the first laboratorymodel cooler to obtain information for the construction of the delivery
model cooler. Results of this evaluation indicated the need for investigating
further the development of a pressure-control device, modifying the model to
provide an easier method of filling, and evaluating coolant container supports
that would be suitable for use in the final model. Therefore, the cooler
model was modified and various pressure control devices were investigated.
Also during this period, solidification techniques for methane were studied
and it was found that solid methane could be produced satisfactorily by the
same methods employed to produce solid nitrogen.

Pressure-control and solidification techniques for neon and hydrogen remain as the main areas for future investigation.

I. WORK ACCOMPLISHED

A. CRYOGENIC-SOLID COOLER, LABORATORY MODEL

A laboratory model of the cryogenic-solid cooler, shown in Figure 1, has undergone tests and evaluation. This model consists of a heat-transfer rod, an inner container for the cryogenic coolant, superinsulation, and an outer jacket which acts as a vacuum bell jar. Four thermocouples were used to measure the temperature distribution along the inner container. One of these thermocouples was attached to the end of the heat transfer rod and the others were mounted at three positions along the outer wall of the coolant container.

In addition to temperature measurements, the pressure over the cryogenic solid, the pressure obtained with the vacuum pump, and the amount of gas evolved from the solid were measured. These measurements were taken before and after steady-state conditions had been established. Both solid methane and solid nitrogen were used as coolants.

The evolution of gas was a measure of the heat leak into the solid coolant, since a known quantity of heat is needed to sublimate a known quantity of solid. These measurements indicated that the heat leak into the solid was 0.41 watts, after equilibrium conditions had been established. Equilibrium conditions were observed six to eight hours after the cooler was filled with the liquid gas. The time required for steady-state conditions was the same, whether the coolant was in liquid or solid form immediately after filling. Both the heat leak value and the time required to attain equilibrium were the same for methane and nitrogen.

The temperature of the heat transfer rod and along the outer wall of the solid container also required from six to eight hours to stabilize. The temperature of the heat-transfer rod under steady-state conditions, agreed within 0.2°K, of the solid temperature measured by its vapor pressure. This temperature remained constant over a period of five days as anticipated and then showed a very sharp increase over a period of a few hours, indicating all the solid had sublimed. The temperature measurements made at intervals along the solid container began to increase after two days; starting with the thermocouple nearest the top of the container. Therefore, a similar array of thermocouples could be used in the final model to give a rough indication of the solid-coolant level in the container.

During the testing of the laboratory model, it became evident that its configuration would have to be modified. There were three reasons for this. First, the model could be filled with cryogenic liquid only after much difficulty; second, tests of various container-support systems proved impractical with this configuration; and third, it was desirable to reduce the heat leak into the solid to a lower value. The third reason listed is most important for ensuring the evaluation of a pressure-control valve under

conditions more closely approximating the parameters expected in the final model. Therefore, it was decided to modify this model in such a manner as to simulate the fill-pump port which would be used in the final model configuration.

The fill-pump port of the laboratory model (Figure 1) was a short metal tube. This tube supported the coolant container, and served as a filling and pumping connection which was far from ideal, and introduced a large heat leak. The first modification consisted of shortening this tube and including an additional vacuum jacket around the coolant container. Superinsulation would then be wrapped around this jacket and the entire assembly would be placed inside the outer vacuum container. The pumping port would consist of a spiral-wound tube attached to the coolant container after the container was filled and before being assembled inside the outer bell-jar. This configuration would permit the coolant container to be filled with ease, reduce the heat leak, and allow support tests to be made. However, it was found that a major problem occurred in providing thermocouple seals through this vacuum jacket. On filling, the seals became cold and cracked, permitting air to enter the insulating vacuum jacket. This modification attempt was discontinued, and a different approach made. This approach consisted of replacing the straight tube fill-pump port with two lengths of thin-wall, 1/8 in. diameter tubing wound as helixes. With this arrangement, the solid coolant container could be filled rapidly from a pressurized storage container. One tube serves as the fill tube and the other as a gas vent. After the container is filled, the liquid may be solidified by pumping through both tubes. The heat leak into the solid coolant was reduced from 0.41 watts to 0.225 watts by replacing the straight tube with the two helical-wound tubes. Also, this configuration permits the investigation of various support techniques for the inner container.

Figures 2, 3, and 4 show the steps in the process of superinsulating of the coolant (inner) container. In Figure 2, the coolant container is mounted in a glass-blowing lathe preparatory to wrapping with superinsulation. The helical tubes can be seen attached to the top of the container. Figure 3 shows the aluminized Mylar and silk being wrapped in alternate layers onto the container. Figure 4 is the insulated container ready to be suspended in the outer vacuum jacket.

B. SOLIDIFICATION TECHNIQUES

In the previous interim engineering report, it was mentioned that solid nitrogen had been produced to within 95 percent of the published density value. The solid was obtained by slowly reducing the pressure over the liquid while it was being mixed. A glass Dewar vessel was used for these solidification experiments.

During this report period, techniques for solidifying liquid methane were investigated. The techniques which have been successful in solidifying liquid nitrogen were applied to methane solidification. The first attempt at solidifying methane required the use of natural gas as a source. The natural gas was reported by the supplier to contain 82- to 92-percent methane, and 4to 10-percent ethane, with other constituents in much lesser proportions. The natural gas was drawn from taps in the laboratory and liquefied in a container immersed in liquid nitrogen. Methane has a boiling point of 111.70K and a freezing point of 90.70K; the other natural gas constituents have melting and boiling points above these values, and thus liquid nitrogen at 77°K should liquefy and freeze the gas. However, it was found that the natural gas mixture remained as a liquid to temperatures slightly above 77°K. Attempts at solidifying the natural gas by reducing the pressure over it were unsuccessful. The liquid had a tendency to foam and bump at reduced pressures in the glass Dewar and this action destroyed any of the solid produced. It was thought that the inability to produce the desired solid in the glass Dewar was due to the large heat leak into this equipment. Therefore, the unmodified version of the laboratory model cooler, having a much smaller heat leak, was filled with liquid (after considerable difficulty due to foaming) and solidification was again attempted. This time it was successful, and a temperature of 68°K was obtained. However, the temperature was not constant, rising slowly and continuously during the several days of the experiment. This was due possibly to a change in concentration of the natural gas as the low boiling point constituents sublimed away from the solid causing a change in concentration. It was thus necessary to abandon natural gas as a source of methane for the solid cryogenic cooler.

A cylinder of CP-grade methane gas, 99.0 percent pure, was ordered and solidification experiments were made. This time, the experiments were successful, and a solid of the expected density was obtained. Both liquid and solid methane from this source behaved in a normal and expected manner in the glass Dewar and the solid cryogenic cooler. Therefore, CP-grade methane, in liquid and solid form, appears to be suitable for use as a cryogenic material in the solid cryogenic cooler.

C. PRESSURE CONTROL

During the past three months, work has continued in the development of a suitable pressure-control valve for the solid cryogenic cooler. Early in the period, efforts to provide a temperature-actuated relief valve were abandoned. The major problem encountered in this valve concept was the high pressure required for sensitivity versus the small valve-movement that could be obtained with available construction materials. It was decided that this type of valve, while desirable in many respects, could not be produced at this time.

Attention was turned to valves which would be sensitive to the difference in the pressure between the external vacuum and the cryogenic container. Two approaches were taken in an effort to solve the pressure-control problem. First, a valve specification was written and sent to ten manufacturers with a quotation request. Three replies were received and it is anticipated that a valve will be ordered during the next reporting period from the vendor who demonstrates the most promising design. Secondly, four experimental control valves were manufactured in the laboratory. These included a ball check-valve, a bellows valve, and two diaphragm valves all of which operate on the difference in pressure between the solid vapor and the external vacuum. Of these, the bellows valve and the valve having a metal diaphragm 0.010-in. thick were determined to be unsatisfactory. However, the ball check-valve, consisting of a nylon ball, a copper seat, and a spring which holds the ball in place, performed satisfactorily. Due to the low available pressure and the small active area of the ball, it was not possible to operate the valve by raising the ball from its seat. However, the force is sufficient for rolling

the ball from its seat which permits the gas to flow from the subliming solid. While not completely satisfactory in its present design, the approach appears to have possibilities of successful operation. The diaphragm valve having a thin metal diaphragm (less than 0.005 in. thick) also operates on the pressure difference between the cryogenic container and the external vacuum. In this case, the area of the diaphragm is sufficiently large to produce the force required for operating the valve. Such a valve is now undergoing evaluation, and preliminary results indicate that a valve of this type, properly designed, is also practical.

D. FINAL MODEL DESIGN

The design of the final laboratory-model cooler has been initiated. Some materials for construction have been received, and the less complex components will be assembled shortly. This model, as now planned, will have a solid container seven inches in diameter and eight inches long. The outer vacuum jacket will be about 14 inches in diameter and 14 inches long with both ends domed for resistance to external pressure. The bellows seals for the fill-pump port have been received and accepted. It is planned to have a removable window over the heat-transfer rod and an evacuation valve to provide a vacuum for the superinsulation. The superinsulation will be three inches thick and the heat leak to the solid container should be not greater than 0.1 watt.

III. CONCLUSIONS AND RECOMMENDATIONS

The modifications of the laboratory model cooler have reduced the heat leak to the cryogenic solid by about one-half. This provides a cooler model for use in evaluating solid-container support and pressure-control techniques under conditions which more closely approximate the heat leak conditions expected of the final model.

The solidification of methane and its use as a cryogenic material in the solid cooler present no problems. Methane has been solidified to the required density through the same techniques developed for the solidification of nitrogen.

The pressure control problem still remains. However, a solution appears to be imminent either through the development of a suitable valve in the laboratory or by a manufacturer.

The design and fabrication schedule for the final model cryogenic-solid cooler is progressing satisfactorily.

Continuation of efforts undertaken during this reporting period is recommended with special emphasis on:

- A. Investigating solidification techniques for hydrogen and neon
- B. Operating the laboratory model with solid hydrogen
- C. Evaluating and selecting a pressure-control device
- D. Designing and developing the final model cooler
- E. Evaluation-testing of this model.

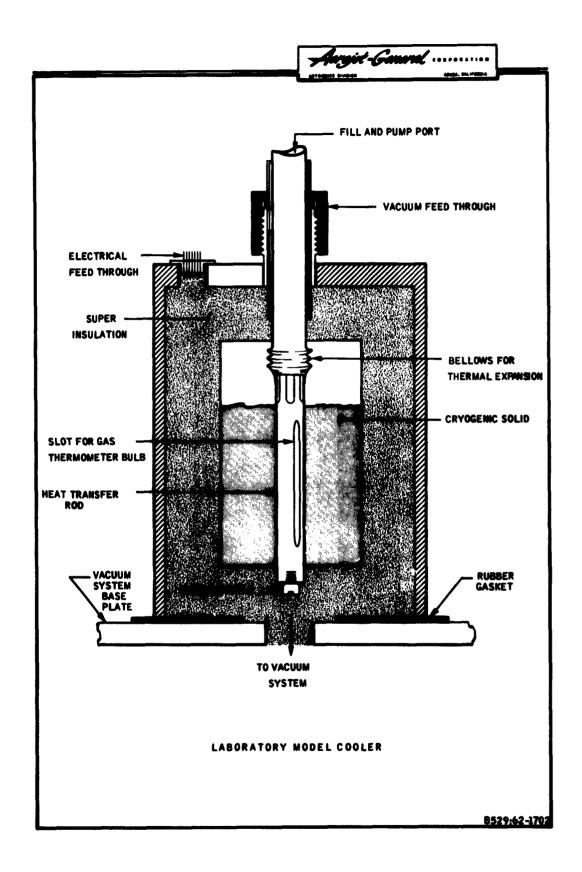




FIGURE 2. COOLANT CONTAINER READY FOR APPLICATION OF SUPERINSULATION MATERIAL



FIGURE 3. SUPERINSULATION WRAPPING SETUP



FIGURE &. SUPERINSULATED COOLANT CONTAINER

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